Filter Component Assessment

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Abstract

In the fall of 1995, commercially available oxide and nonoxide-based monolithic and advanced fiber reinforced candle filters were installed in the Westinghouse Advanced Particulate Filtration (APF) system at the Foster Wheeler pressurized circulating fluidized-bed combustion (PCFBC) test facility in Karhula, Finland, and were operated for a maximum of 626-1166 hours at temperatures of 850°C. The response and performance of the porous ceramic filter materials to extended operation in the high temperature, oxidizing environment, and information pertaining to the chemistry and morphology of the dust cake deposits formed throughout the filter cluster assembly during testing in 1995-1996 are presented in this paper.[†]

As a result of testing at Karhula, the oxide-based Coors P-100A-1 alumina/mullite candle filters continued to show promise for achieving extended operating life in advanced high temperature coal-fired applications. Additional testing in the high temperature PCFBC oxidizing environment is, however, essential for demonstration of the viability of the high temperature, creep resistant, clay bonded silicon carbide Schumacher Dia Schumalith FT20 and Pall 326 candle filters, as well as the 3M CVI-SiC composite filter elements.

Efforts at Westinghouse were also focused on qualifying several advanced filter elements for possible installation and operation in Foster Wheeler's PCFBC test facility in the fall of 1997. As part of the DOE initiatives, these elements included the oxide-based Nextel™ 610 and 720 continuous fiber reinforced ceramic composite filters manufactured by 3M, B&W, and Techniweave, and the modified, oxide-based, filament wound DuPont PRD-66 candles with expected enhanced membrane integrity and barrier filtration characteristics. Additional elements that were subjected to qualification testing included Blasch's newly developed mullite-bonded alumina monolithic candles, Specific Surface's Taperflow cordierite-based monolithic filters,

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and Scapa Filtration's vacuum infiltrated oxide-based fibrous candles. Qualification testing at Westinghouse included concurrent exposure of the elements to ~120 hours of steady state testing under simulated pressurized fluidized-bed combustion (PFBC) conditions, ~2200 accelerated pulse cleaning cycles, and 12 thermal transient events in order to demonstrate not only the performance and viability of the various elements, but also the thermal and initial chemical stability of the filter matrices, and the mechanical integrity of the filters in retrofit applications. Preliminary results of this effort are presented in this paper.

Westinghouse has also been involved in the design and construction of a slipstream filter vessel that will be installed and operated at the Sierra Pacific Power Company, Piñon Pine, Tracy No. 4 Station in Reno, Nevada. Commercially available and advanced mini-candles will be installed in the filter array, and subjected to long-term exposure under particle-free Integrated Gasification Combined Cycle (IGCC) operating conditions. Additional porous ceramic filter and structural metal coupons will be exposed to the fuel gas environment in a flow-over manner.

Introduction

Since the late 1970's development and utilization of porous ceramic filter elements have expanded from the manufacture of monolithic oxide-based and clay bonded silicon carbide candles to the production of filament wound, continuous fiber reinforced ceramic composite, and sintered metal candle filters. Similarly transitioning of the candle filter technology to the production of monolithic cross flow filters, as well as alternate geometric filter configurations has occurred.

Emphasis at Westinghouse since 1990 has been focused on the operation of APF systems in which 384 commercially available, 1.5 m porous ceramic candle filters were subjected to PFBC conditions at the American Electric Power (AEP) Tidd Demonstration Plant in Brilliant, Ohio. Similarly, 128 commercially available, 1.5 m candles were installed and operated in Westinghouse's APF at the Foster Wheeler PCFBC pilot-scale test facility in Karhula, Finland. The total operating service life of surveillance filters which have been installed at both test facilities is shown in Table 1. Opportunities to continue testing commercially available monolithic elements, and the introduction and utilization of alternate monolithic and advanced continuous fiber reinforced ceramic composite candles is currently planned in Westinghouse's APF systems at Karhula and at the Southern Company Services test facility in Wilsonville, Alabama, as well as in alternate supplier's filtration systems at several pilot-scale test facilities throughout the country.

Achieving three years of operational filter element life is the primary goal of the Westinghouse hot gas filtration programs. Efforts to demonstrate viable operation of the plant and warranted filter life are currently the focus of the Clean Coal Program being undertaken at the McIntosh Unit 4 Demonstration Plant in Lakeland, Florida, where 1500-3000 candle filters will be subjected to PFBC and carbonizer conditions. Similarly installation of 748 candle filters has been completed in the Westinghouse APF system at the Sierra Pacific Power Company, Piñon Pine, Tracey No. 4 Station in Reno, Nevada, with commissioning of the IGCC Demonstration Plant targeted in 1997.

Table 1
Westinghouse APF Field Experience

Filter Supplier/ Matrix	Maximum Operating Hours	
	American Electric Power Brilliant, Ohio PFBC	Foster Wheeler Karhula, Finland PCFBC
Schumacher F40	5855	227[(a)]
Schumacher FT20	1705	1620[(b)]
Pall Vitropore 442T	1705	1341[(a)]
Pall 326	—	1620[(b)]
Coors P-100A-1 Alumina/Mullite	2815	716[(a)] 1620[(b)] 2730[(c)]
3M CVI-SiC	1705	627[(d)]
DuPont PRD-66	1705	—

(a) 1992-1994 test campaign.
(b) 1995-1997 test campaign.
(c) 1620 hrs of operation under PCFBC conditions at Karhula and 1110 hrs of operation under PFBC conditions at AEP.
(d) 1995-1996 test campaign.

Prior to installation of newly developed candles in the field, Westinghouse subjects several as-manufactured filter elements to a simulated PFBC environment in a qualification test program conducted in Pittsburgh, Pennsylvania. An assessment of the retrofit compatibility of the candles with existing hardware, utilization of appropriate mounting and sealing techniques, general filtration performance characteristics of the elements, and thermal and initial chemical stability of the filter matrices is easily obtained during performance of the qualification program.

As in previous field test surveillance programs conducted by Westinghouse,[1,2] both the as-manufactured and qualification-tested candles undergo extensive nondestructive and destructive evaluation in order to demonstrate whether possible changes within the filter matrix result which impact operating life. This effort generally includes determination of the room temperature and process operating temperature bulk strength of the filter matrix, as well as ultimate load bearing capability, burst pressure, hoop stress, elastic modulus, and Poisson's ratio. The as-manufactured material properties which have been developed by Westinghouse for both the monolithic and advanced ceramic composite filter elements are shown in Table 2. For consistency, 15 mm c-ring sections are removed from each candle filter for use in the room temperature and process temperature bulk strength analyses, while ~25 cm sections are removed from each candle for use in the burst strength characterization.

Concern has been raised as to the validity of the reported bulk strength of the composite materials when 15 mm c-rings are utilized. Table 3 demonstrates that little variation in the reported strengths of the 3M CVI-SiC candle results when either 15 mm or 25 mm c-ring or

Table 2
Material Properties of the As-Manufactured Monolithic and Advanced Porous Ceramic Filters

Filter Matrix (15 mm)	25°C C-Ring Strength, psi		25°C C-Ring Ultimate Load, lbs		Burst Pressure, psi	Ultimate Hoop Stress, psi	Elastic Modulus, psi x 10^6	Poisson's Ratio
	Compression	Tension	Compression	Tension				
Coors P-100A-1 Alumina/Mullite	2575±182	2721±415	42.8±2.4	31.4±4.9	860	2317	5.7	0.23
Blasch 4-270 Mullite Bonded Alumina	625±148	656±92	13.3±3.6	9.2±1.5	170	410	2.1	0.09
Specific Surface Taperflow™	509±106	638±198	2.2±0.2	2.1±0.6	ND	ND	ND	ND
Schumacher Dia Schumalith F40	1300±213 (a) 1790±112 (b)	1907±111 2308±275	74.5±14.2 102.1±9.4	53.4±3.7 63.8±8.2	1140-1370	1893-2267	5.89-6.26	0.25-0.31
Schumacher Dia Schumalith FT20	2296±261	2268±167	44.2±4.8	29.2±2.9	665	1703	7.3	0.17
Pall Vitropore 442T	2857±186	2574±177	60.5±5.9	35.3±1.8	513-970	1307-2499	5.25-6.67	0.14-0.19
Pall 326	2961±186	2480±124	60.7±3.9	32.6±0.7	ND	ND	ND	ND
DuPont PRD-66	1219±162	1265±188	9.2±1.0	7.3±1.3	120	462	9.36	2.25

ND: Not determined.
NR: Not reported.
(a) 1991 Production lot.
(b) 1992 Production lot.
(c) Composite strength (Outer confinement layer, filtration mat, and triaxial support braid).
(d) Triaxial support braid.
(e) Seam rupture.
* 15 mm o-ring diametral compressive strength or load.

Table 2 (Continued)
Material Properties of the As-Manufactured Monolithic and Advanced Porous Ceramic Filters

Filter Matrix (15 mm)	25°C C-Ring Strength, psi		25°C C-Ring Ultimate Load, lbs		Burst Pressure, psi	Ultimate Hoop Stress, psi	Elastic Modulus, psi x 10^6	Poisson's Ratio
	Compression	Tension	Compression	Tension				
3M CVI-SiC Composite	1903±362 (c) 12448±2620 (d)	1715±281 (c) 10718±2035 (d)	2.1±1.0	2.1±0.4	NR	1.01 ksi	2.96-3.38	0.14-0.27
DuPont SiC-SiC	11905±1556 16303±1249 *	20210±4139	4.12±0.38 18.57±2.18 *	6.48±0.84	ND	2972 (e)	ND	ND
3M Oxide-Based CFCC	483±162 (c) 1929±648 (d)	624±154 (c) 2389±590 (d)	0.41±0.14	0.50±0.13	52	586	1.36	0.73
B&W Oxide-Based CFCC	547±58	675±91	2.4±0.2	2.3±0.3	188	998	1.25	0.95
Techniweave 610 Oxide-Based CFCC	2324±548	1190±233	7.7±1.5	3.3±1.3	ND	ND	ND	ND
Techniweave 720 Oxide-Based CFCC	2215±431	1377±413	6.2±0.6	3.1±0.9	ND	ND	ND	ND
IF&P Fibrosic™	28.1±6.7 53.2±5.5 *	50.9±12.0	0.64±0.12 2.6±0.2 *	0.71±0.15	ND	41	0.5	0.4
Scapa Cerafil™	177±24	187±32	3.5±1.3	2.6±0.4	ND	ND	ND	ND

ND: Not determined.
NR: Not reported.
(a) 1991 production lot.
(b) 1992 production lot.
(c) Composite strength (Outer confinement layer, filtration mat, and triaxial support braid).
(d) Triaxial support braid.
(e) Seam rupture.
* 15 mm o-ring diametral compressive strength or load.

Table 3
Bulk Strength of the As-Manufactured 3M CVI-SiC Composite Matrix

Test Methodology	Sample Width, mm	Test Temperature, °C	Composite[(a)] Strength, psi	Support Braid[(b)] Strength, psi
O-Ring Diametral	15	25	1315±186	13276±1417
O-Ring Diametral	15	732	1104±350	11250±3595
O-Ring Diametral	25	25	1314±254	14026±2012
O-Ring Diametral	25	732	1060±219	11012±1795
C-Ring Compression	15	25	1343	13187
C-Ring Compression	15	732	1352	13444
C-Ring Compression	25	25	1635	17012
C-Ring Compression	25	732	1425	16857

(a) Calculated based on the entire thickness of the as-received filter wall.
(b) Calculated based on the thickness of the triaxial braid layer.

o-ring sections are used to determine the bulk strength of the composite filter matrix. In addition to determination of the bulk strength of the various filter materials, changes in the microstructure, fracture toughness, and phase composition are determined via scanning electron microscopy/ energy dispersive x-ray analysis (SEM/EDAX) and x-ray diffraction analysis (XRD).

As a result of qualification testing, modifications of the ceramic matrix and/or component construction for extended service life can be quickly identified prior to use in the field. Several of the matrix or design modifications identified by Westinghouse have included

- Identifying the need to achieve acceptable dimensional tolerance specifications of the as-manufactured filter elements in order to assure retrofit and ease of removal from existing system hardware
- Attaining acceptable as-manufactured and post-test gas flow resistance
- Modification of the outer surface of the filter element to assist dust cake removal
- Densification of the flange and/or end cap primarily for enhanced strength
- Manufacture of the 1.5 m candle filter as an integral unit (i.e., absence of lapped seams or butt joints, mold discontinuities, inserted plugs, stacked plates, etc.)
- Selection of an appropriate membrane to permit barrier filtration as opposed to bulk filtration
- Selection of an appropriate fiber and/or architecture that minimizes embrittlement while maximizing strength and load bearing capabilities
- Selection of an appropriate thickness of the deposited CVI-SiC layers along fiber preforms in order to limit oxidation and/or removal of the nonoxide layers during PFBC/PCFBC operation

- Utilization of an oxide matrix or binder in continuous fiber reinforced ceramic composite filters to prevent external and internal surface oxidation of the deposited CVI-SiC layers. Oxidation typically results in bonding of the encapsulating shells to the underlying fiber(s), leading to embrittlement or reduced fracture toughness of the matrix
- Utilization of an oxide matrix with negligible free silica in order to prevent the formation of low melting temperature silicate eutectics, particularly in gas phase alkali-enriched process gas environments.

In addition to component design and matrix modifications, qualification testing has permitted Westinghouse to develop the appropriate mounting hardware to accommodate variations in the flange contour that generally result from production constraints that are inherent to the filament winding and fiber preforming processes.

Once qualified in terms of retrofit and simulated PFBC performance, production, installation, and operation of the elements in the field is undertaken at a perceived reduced risk for failure. Commercial production of homogeneously manufactured elements in quantities of 500-3000 candles annually is viewed by Westinghouse as an additional critical requirement for viable operation of advanced hot gas filtration systems.

From previous field experience, Westinghouse has demonstrated that with respect to the oxide-based monolithic filter elements as the Coors P-100A-1 alumina/mullite candle, crystallization of the matrix typically occurs as a response of the material to the high temperature process environment. In addition, potential failure of the oxide-based monolithic filter elements may occur due to thermal fatigue of the matrix under abnormal operating conditions. For the nonoxide monolithic materials as the clay bonded silicon carbide Schumacher and Pall filter elements, enhanced creep resistance of the original Dia Schumalith F40 and 442T materials, respectively, was required to prevent elongation and/or creep crack growth, and ultimately failure of the elements. With improvements in the binder systems of both materials, high temperature creep resistant binders were developed. Westinghouse introduced the use of the high temperature creep resistant Schumacher Dia Schumalith FT20 and Pall 326 candles in test programs conducted at Karhula in 1995-1996.[3] With extended exposure in the PCFBC environment, elongation of the elements was shown to continue to occur, but at a reduced rate in comparison to the originally formulated materials. Mechanisms were postulated which implied that oxidation of the silicon carbide grains was primarily responsible for uniform volume expansion of the clay bonded silicon carbide filter elements during the initial 1000-2000 hours of operation in the PCFBC environment. Continued exposure, however, is needed to demonstrate whether a conditioned level of oxidation is attained with time, or if high temperature creep will occur.

Similar to the clay bonded silicon carbide candles, oxidation and/or removal of the 2 μm chemically vapor infiltrated silicon carbide (CVI-SiC) layer resulted along the outer confinement and filtration mat layers of the 3M composite filter element. Increasing the thickness of the deposited CVI-SiC layer, addition of an oxidation resistant coating, or utilization of an oxide

matrix as a replacement for the deposited CVI-SiC were considered as potential options for improving the stability of the nonoxide composite filters.

Although improvements to the material properties and matrix composition can be made, and component design modifications can be implemented, successful long-term operation of the advanced hot gas filtration system also requires

- Eliminating the occurrence of ash bridge formation within the filter arrays
- Efficient removal of the dust cake layer along the surface of the filter elements
- Minimizing particle reentrainment
- Preventing deposition and/or condensation of volatile material throughout the filter matrix.

In addition, the relationship of ash or char particle size, composition, and sorbent to coal feed ratios for use at system design temperatures and pressures, the long-term stability of the porous ceramic materials or sintered metals, system design and stability of the structural metals, and operation of the combustion or IGCC plant, are all critical issues that concurrently impact successful extended operation of the advanced particulate filtration systems.

Objectives

The objectives of the Filter Component Assessment program are to

- Provide a more “ruggedized” filter system that utilizes porous ceramic filters which have improved resistance to damage resulting from crack propagation, thermal fatigue and/or thermal excursions during plant or process transient conditions, and/or mechanical ash bridging events within the candle filter array (Task 1).

- Assess the effects of long-term (i.e., 1000-1500 hours) pilot-scale exposure under actual pressurized circulating fluidized-bed combustion (PCFBC) conditions on advanced candle filter failure modes and degradation mechanisms (Task 2).

- Assess the stability of select advanced filter materials when subjected to long-term exposure in actual integrated gasification combined cycle (IGCC) gas streams (Task 3).

Project Description

Initially effort in Task 1 of the Filter Component Assessment program was focused on evaluating the filtration characteristics, mechanical integrity, and corrosion resistance of the following second generation candle filters for use in advanced coal-fired applications:

- 3M CVI-SiC composite — Chemical vapor infiltration of silicon carbide on an aluminosilicate Nextel™ 312 triaxial braid preform, on a chopped alumina-enriched filtration mat layer, and on an open mesh Nextel™ 312 outer confinement layer
- DuPont PRD-66 — Filament wound candle containing corundum, cordierite, cristobalite, and mullite
- DuPont SiC-SiC composite — Chemical vapor infiltration of silicon carbide on a Nicalon™ felt or mesh screen support layer
- IF&P Fibrosic™ — Vacuum infiltrated, oxide-based, chopped fibrous matrix.

Recently this effort has been expanded to include the following additional advanced monolithic and continuous fiber reinforced ceramic composite candle filters:

- 3M oxide-based composite — Infiltration of an aluminosilicate phase on an aluminosilicate Nextel™ 610 or Nextel™ 720 triaxial braid preform, on a chopped alumina-enriched filtration mat layer, and on an open mesh Nextel™ 610 or Nextel™ 720 outer confinement layer
- DuPont PRD-66 — Filament wound candle containing corundum, cordierite, cristobalite, and mullite (Improved outer surface membrane)
- B&W oxide-based composite — Vacuum wound element containing continuous Nextel™ 610 fibers, chopped Saffil fibers, and an alumina-enriched sol-gel binder
- Techniweave oxide-based composite — Continuously woven 2D Nextel™ 720 or Nextel™ 610 matrix infiltrated with a mullite sol. A 3D reinforcement is added to prevent delamination.
- Blasch 4-270 — Injection molded, monolithic, mullite-bonded alumina matrix
- Scapa Cerafil™ — Vacuum infiltrated, oxide-based, chopped fibrous matrix
- Specific Surface Taperflow™ — Monolithic cordierite matrix.

Qualification testing in Westinghouse's pressurized fluidized-bed combustion test facility in Pittsburgh, PA, was utilized to demonstrate retrofit capabilities of the above elements into existing system hardware, as well as high temperature filtration characteristics, mechanical integrity, and general operating performance of the various advanced candle filters. Testing was conducted for a period of ~120 hours at steady state operating conditions. Subsequently the filter array was subjected to ~2200 accelerated pulse cleaning cycles in order to demonstrate the viability of the various matrices to extended thermal fatigue, and finally 12 thermal transients which simulated rapid cooling and/or heating of the array during plant excursions. Currently the residual bulk strength, fracture toughness, composition, and microstructure of each advanced filter matrix is being characterized. Where warranted, modifications to the manufacturing process have been identified and conveyed to each supplier in order to produce filter elements that do not contain seams, joints, plugs, or attached sections, but which achieve the required dimensional tolerances and design specifications, acceptable initial gas flow resistance and bulk matrix strength for use during extended operation in advanced coal-fired process applications.

In order to assess the effects of long-term pilot-scale exposure on advanced candle filter failure modes and degradation mechanisms in Task 2,

- Advanced monolithic, high temperature, creep resistant Schumacher Dia Schumalith FT20 candles
- Advanced monolithic, high temperature, creep resistant Pall 326 candles
- Coors P-100A-1 alumina/mullite candles
- 3M CVI-SiC composite candles

were installed in the Westinghouse APF system that was operated at Foster Wheeler's PCFBC test facility in Karhula, Finland. Three test campaigns were completed between November 1995 and October 1996 in which 112-128 candles were operated for a period of 1166 hours at temperatures of ~850ºC. Illinois No. 6 coal was used as the feed material, and either Linwood or Iowa Industrial limestone were utilized as sulfur sorbents.

Post-test characterization of PCFBC-exposed surveillance candles included determining the resulting gas flow resistance of the filter elements, residual process temperature bulk strength, high temperature creep and thermal expansion properties, and residual microstructure and phase composition. In addition, characterization of ash samples that were removed from various locations within the three filter arrays was also conducted. These analyses included determining the bulk density, moisture content, bulk strength, thermal expansion, and identification of the morphology and composition of the ash materials.

PCFBC testing was reinitiated in April 1997, and continued through May 1997, during which time Beech Fork coal and Gregg limestone were utilized as the feed and sulfur sorbent, respectively. Approximately 454 hours of operation of the Westinghouse APF were completed during this test segment. The 128 candle filter array which was operated at temperatures of ~850°C consisted of a mixture of newly manufactured, as well as previously PCFBC or PFBC/PCFBC-exposed Schumacher Dia Schumalith FT20, Pall 326, and Coors P-100A-1 alumina/mullite candle filters. A second test campaign is currently planned to begin in August 1997, during which time the Westinghouse APF will operate for approximately an additional 500 hours at temperatures of 760°C. In this test campaign, the advanced oxide-based monolithic and composite candles filters are targeted to be installed and operated in conjunction with previously PCFBC-exposed Schumacher Dia Schumalith FT20, Pall 326, and Coors P-100A-1 alumina/mullite candle filters.

Additional effort has been focused in Task 3, to assess the stability of select advanced filter materials during long-term exposure in actual IGCC gas streams. To date design of a pressurized mini-vessel system has been completed, its position within the Sierra Pacific Power Company, Piñon Pine, IGCC Demonstration Plant in Reno, Nevada, has been identified, mini-candle filter elements have been procured, and metal structural coupons for exposure in the mini-vessel have been acquired. Construction and supply of the unit will be initiated after approval of the Westinghouse design specification packages for the pressure vessel and pulse gas skid have been received from Sierra and M. W. Kellogg, and interface engineering has been completed by M. W. Kellogg for retrofit of the unit into the existing plant.

Results and Accomplishments

The following sections discuss the results and highlight the accomplishments that have been made by Westinghouse during conduct of the Filter Component Assessment program and associated Advanced Hot Gas Filter Development PRDA programs throughout the past year.

Assessment of PCFBC Field-Exposed Advanced Candle Filters[4]

Hot gas filtration testing was initiated in the Westinghouse APF in November 1995 at the Foster Wheeler PCFBC test facility in Karhula, Finland. Three test campaigns were conducted utilizing 1.5 m high temperature, creep resistant, clay bonded silicon carbide Schumacher Dia Schumalith FT20 and Pall 326 candle filters in the top and middle arrays, respectively, and a mixture of the 1.5 m Coors P-100A-1 alumina/mullite and 1.5 m 3M CVI-SiC composite filter elements in the bottom array (Table 4). In addition, five Coors P-100A-1 alumina/mullite candle filters which had been operated for a period of 1110 hours in the Westinghouse APF system at the AEP PFBC Tidd Demonstration Plant in Brilliant, Ohio, were installed in the bottom array. A total of 1166 hours of hot gas filtration testing was achieved during the 1995-1996 test program in Karhula, Finland.

After each test campaign, surveillance filter elements were removed from the cluster and were subjected to both nondestructive and destructive materials characterization in order to identify the overall integrity of each element, as well as to determine the residual strength of the various filter matrices at process operating temperature, and any microstructural changes that may have occurred as a result of operation in the PCFBC environment. Additional analyses were focused on determining the residual high temperature creep characteristics of the PCFBC-exposed clay bonded silicon carbide filter elements, their potential to undergo oxidation, and the associated impact of ash and ash chemistry on the stability of the various filter matrices. The results of these efforts are presented in the following sections.

Room Temperature Gas Flow Resistance

The gas flow resistance of the as-manufactured Schumacher Dia Schumalith FT20, Pall 326, Coors P-100A-1 alumina/mullite, and 3M CVI-SiC composite candle filters is shown in Figure 1. All as-manufactured candle filters initially met the Westinghouse room temperature gas flow resistance tolerance of $\leq$1.0 in-wg/fpm.

Similar gas flow resistance measurements were obtained for the PCFBC surveillance filter elements that were returned to Westinghouse at the conclusion of Test Segments 2 and 3. A significant increase in the room temperature gas flow resistance across each element resulted after testing at Karhula (Figure 2). The nature and thickness of the ash cake layer that remained along the outer surface of each candle typically governed the resulting gas flow resistance of each PCFBC-tested filter element.

Table 4
Summary of PCFBC Testing

Test Segment	1 (11/95-12/95)	2 (2/96-4/96)	3 (8/96-10/96)
Coal	Illinois No. 6 (Sparta)	Illinois No. 6 (Sparta)	Illinois No. 6 (Sparta)
Sorbent	Linwood Limestone	Linwood Limestone	Linwood Limestone Iowa Industrial Limestone Resized Linwood Limestone
Number of Candles	112	112	128
Schumacher FT20	32-35	35	46
Pall 326	32-35	35	45
Coors Alumina/Mullite	24 (5) - 42 (5)[(a)]	33 (5)[(a)]	32 (4)[(a)]
3M CVI-SiC Composite	24-0	9	5
Operating Hours (Coal)	153[(b)]	387	626
Operating Temperature, °C	826-853	818-860	838-860
Operating Pressure, bar	10.7-11.1	10.6-11.3	10.5-10.7
Inlet Dust Loading, ppmw	12,000-13,500	12,000-15,500	11,000-12,500
d_{50}, µm (Malvern)	NA	NA	23 (20-26)

(a) Number of installed PFBC-exposed Coors filter elements (AEP Test Segment 5) shown in parentheses.
(b) Thirty-five hours of initial operation prior to removal of the 3M CVI-SiC composite filters, followed by 118 hours of continuous operation.
NA: Not available.

Compressive and Tensile Strength

Strength characterization of the as-manufactured Schumacher Dia Schumalith FT20, Pall 326, Coors P-100A-1 alumina/mullite, and 3M CVI-SiC composite candle filters was conducted via compressive and tensile testing of 15 mm c-ring sections at room temperature, 850°C, and 900°C. In addition, surveillance filters were removed after completion of each test segment, and were subjected to c-ring compressive and tensile strength testing at room temperature, 850°C, and 900°C.

The as-manufactured Schumacher Dia Schumalith FT20 and Coors P-100A-1 alumina/mullite filter materials retained their brittle fracture characteristics during c-ring compressive and tensile testing up to temperatures of 900°C. In contrast, the as-manufactured Pall 326 filter matrix retained its brittle fracture characteristics up to temperatures of 850°C, but experienced plastic deformation at temperatures of 870°C and 900°C. The composite fracture characteristics of the as-manufactured 3M CVI-SiC filter matrix were retained at all test temperatures.

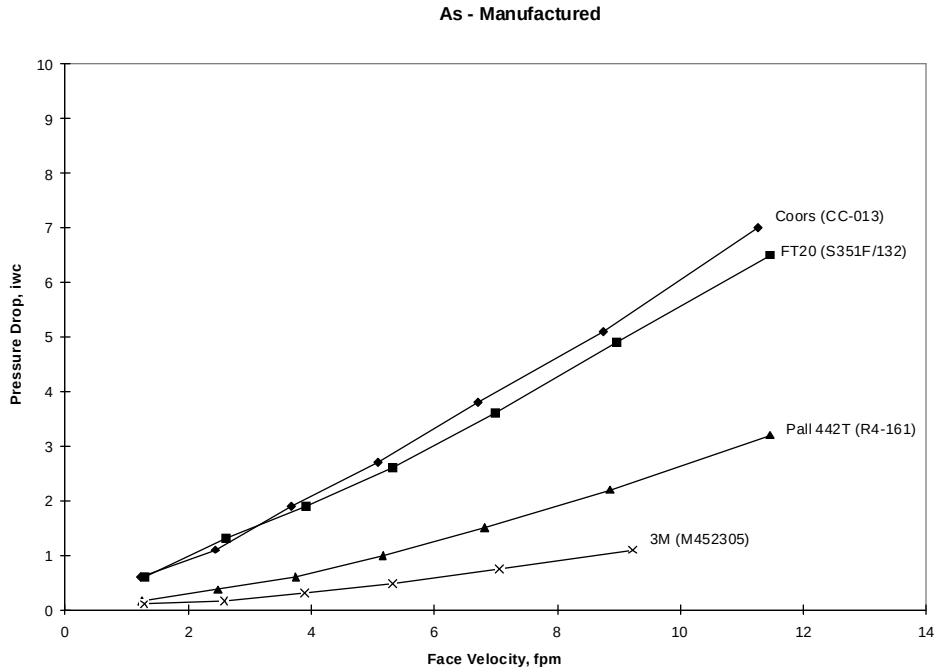

Figure 1 — Room temperature gas flow resistance of the as-manufactured candle filters.

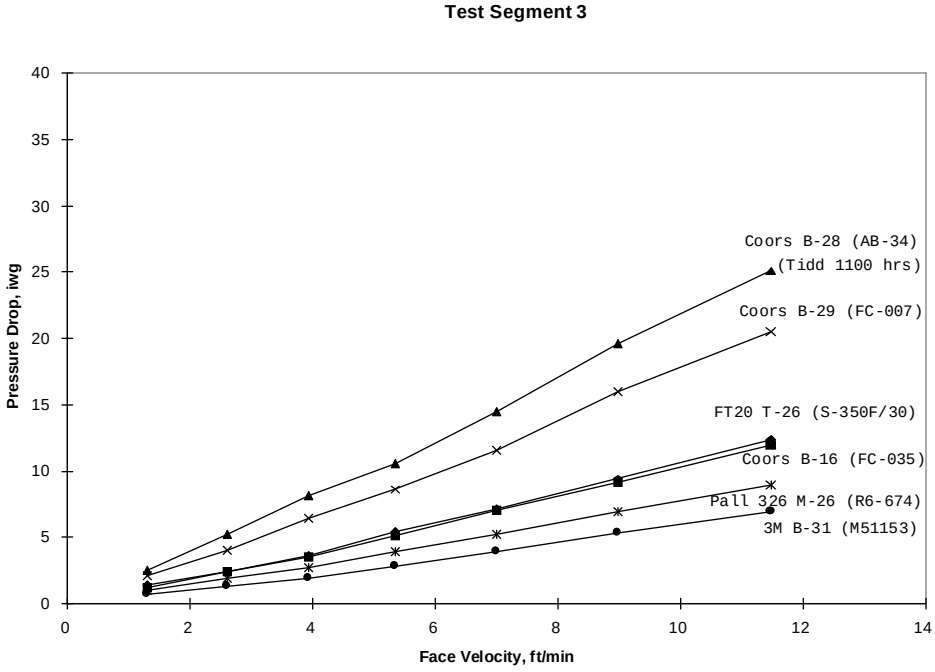

Figure 2 — Room temperature gas flow resistance of the surveillance candle filters that were removed from the Westinghouse APF cluster assembly at the conclusion of Test Segment 3.

After 1166 hours of operation in the PCFBC environment, the c-ring compressive and tensile strengths of the Schumacher Dia Schumalith FT20 matrix tended to increase (Figures 3 and 4). In contrast the Pall 326 matrix initially experienced a loss of bulk matrix c-ring compressive and tensile strength, but achieved a conditioned compressive strength with extended process operation. The Coors P-100A-1 alumina/mullite matrix tended to gradually lose c-ring compressive strength as reflected by both the PCFBC-exposed and PFBC Tidd-exposed filter elements. In contrast, an initial loss of c-ring tensile strength resulted, followed by a conditioning and gradual increase in bulk strength of the Coors P-100A-1 alumina/mullite filter matrix. The 3M CVI-SiC composite matrix initially experienced a loss of c-ring compressive and tensile strength, followed by a conditioning or stabilization of the matrix during extended process operation.

Although operation in the PCFBC environment led to changes in the bulk strength of each porous ceramic filter matrix, a conditioned compressive or tensile strength of 1000-3500 psi was sufficient for continued operational viability of the commercially available monolithic and second generation fiber reinforced candle filters in advanced coal-fired applications.

Hoop Stress, Elastic Modulus, and Poisson's Ratio

An ~25 cm section of material was removed from each of the Test Segment 2 and Test Segment 3 PCFBC-exposed surveillance candle filters. Two 90° strain gage rosettes were installed along both inside and outside surfaces of the filter sections, at approximately the center of each test sample. A water filled bladder was inserted into the i.d. bore of each filter section, and was subsequently pressurized to determine the ultimate hoop strength of the filter material. The outer strain gage measurements for the 3M CVI-SiC composite filter material were considered to be somewhat in question, since delamination and shear were expected to have resulted between the layers of the composite structure during burst testing.

The pressure required to fail each filter section, the ultimate hoop stress, elastic modulus, and Poisson's ratio for the PCFBC-exposed surveillance filter sections are presented in Table 5. Also included in Table 5 are similar material properties for the as-manufactured filter materials.

High Temperature Creep

High temperature creep testing was conducted on 115 mm x 8.5 mm x 12 mm bars that were removed from the 540 hour, PCFBC-exposed, Schumacher Dia Schumalith FT20 and Pall 326 candle filters. The Schumacher Dia Schumalith FT20 and Pall 326 filter materials unlike the Schumacher Dia Schumalith F40 and Pall Vitropore 442T filter materials, did not exhibit high temperature creep when a 500 psi, 4-point bend, flexural load was applied to the surface of the bend bars for a period of ~300-500 hours at temperatures of 843°C. The enhanced high temperature creep resistance of the Schumacher Dia Schumalith FT20 and Pall 326 filter matrices resulted from manufacturing changes that had been made to the binder phase during production of both filter elements.

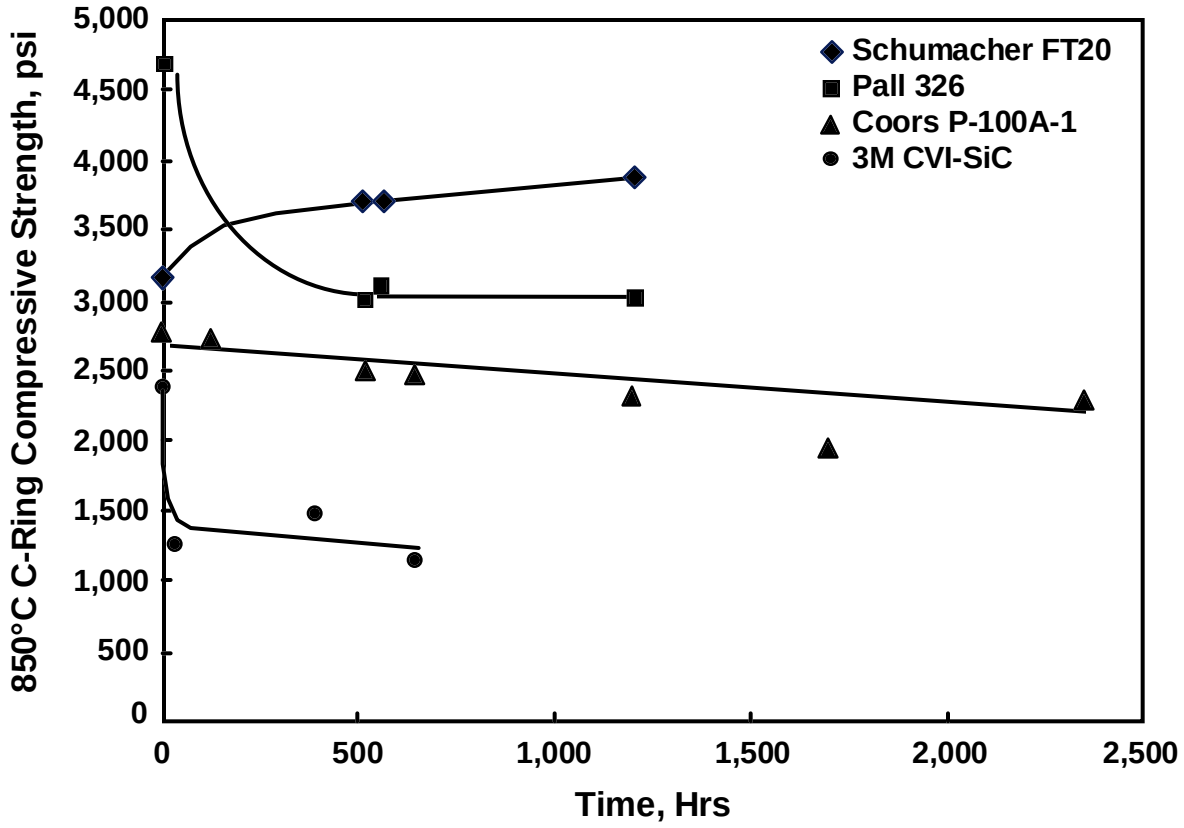

Figure 3 — Residual process temperature compressive strength of the PCFBC-exposed candle filters.

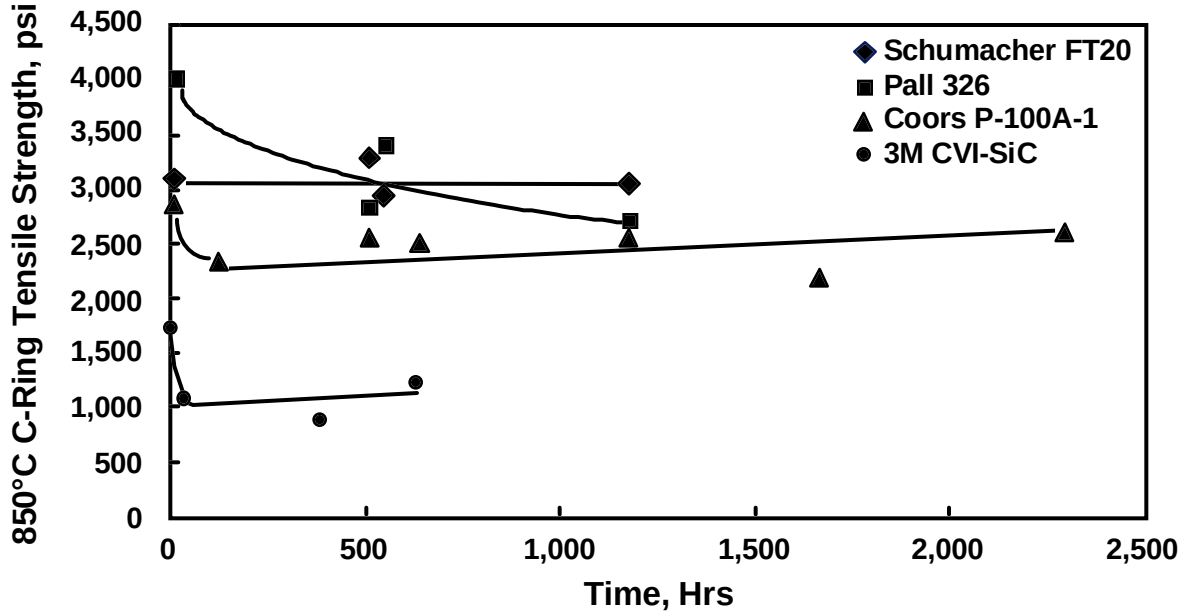

Figure 4 — Residual process temperature tensile strength of the PCFBC-exposed candle filters.

Table 5
Material Properties of the PCFBC-Exposed Porous Ceramic Candle Filters
—Test Segments 2 and 3 —

Candle Identification Number	Operating Time, Hrs	Burst Pressure, psi	Ultimate Hoop Stress, psi	Modulus, psi x 10^6	Poisson's Ratio
Schumacher Dia Schumalith FT20					
S350F/108	—	665	1703	7.3	0.17
S350F/8 (T12)	540	555	1496	7.44	0.21
S350F/42 (B15)	505	590	1584	7.39	0.15
S350F/30 (T26)	1166	720	1942	5.77	0.11
Pall 326					
R3-676	—	ND	ND	ND	ND
R5-655 (M21)	540	525	1369	5.00	0.16
R5-654 (B21)	505	520	1344	5.15	0.16
R6-674 (M26)	1166	650	1641	4.83	0.13
Coors P-100A-1 Alumina/Mullite					
FC-030	—	860	2317	5.7	0.23
FC-070 (B22)	505	540	1503	4.84	0.21
DC-051 (B1)	1650[(a)]	505	1373	5.18	0.20
FC-035 (B16)	626	520	1425	3.90	0.18
FC-007 (B29)	1166	565	1402	3.44	0.25
EC-014 (B28)	2276[(a)]	505	1380	4.37	0.24
3M CVI-SiC Composite					
M-51076	—	ND	1.01 ksi	2.96-3.38	0.14-0.27
M-51103 (B36)	387	133	1179	3.35	0.22
M-51153 (B31)	626	105	946	5.59	0.34

ND: Not determined.
(a) PFBC/PCFBC-exposed candle filter.

High temperature creep testing was similarly conducted on the 1166 hour, PCFBC-exposed, Schumacher Dia Schumalith FT20 and Pall 326 filter materials, and on the 2276 hour, PFBC/PCFBC Coors P-100A-1 alumina/mullite filter material. When a load of 500 psi was applied to the surface of the bend bars, virtually no or negligible creep strain was identified after ~500 hours (Table 6).

Similar high temperature creep testing was conducted using the 387 hour, PCFBC-exposed, 3M CVI-SiC composite filter material. Although delamination of the triaxial braid from the filtration mat and outer confinement layers resulted, testing indicated that the 3M CVI-

Table 6
High Temperature Creep Strain

Filter Material	AEP-PFBC		FW-PCFBC		HT Creep Testing			Total Creep Strain
	Hrs	Temp, °C	Hrs	Temp. ºC	Load, psi	Temp, °C	Hrs	Percent
Schumacher Dia Schumalith F40	—	—	—	—	500	843	18-53	1.47-2.03[a]
Schumacher Dia Schumalith F40	3038	732	—	—	500	843	114	0.42
Schumacher Dia Schumalith FT20	—	—	—	—	500	843	300	0.085[a]
Schumacher Dia Schumalith FT20	—	—	540	850	500	843	500	0
Schumacher Dia Schumalith FT20	—	—	1166	850	500	850	510	0.038
Pall Vitropore 442T	—	—	—	—	500	843	300	2.81[a]
Pall Vitropore 442T	—	—	1341	830	500	843	141	3.41
Pall 326	—	—	—	—	500	850	500	0.085[a]
Pall 326	—	—	540	850	500	850	500	0
Pall 326	—	—	1166	850	500	843	510	0
Coors P-100A-1 Alumina/Mullite	—	—	2276[b]	850	500	850	510	0.019

(a) Significantly reduced creep strain for the high temperature creep resistant Schumacher Dia Schumalith FT20 and Pall 326 filter materials in comparison to the Schumacher Dia Schumalith F40 and Pall Vitropore 442T filter materials.
(b) PFBC/PCFBC-exposed filter element.

SiC composite matrix did not exhibit creep strain after ~300-500 hours when a 500 psi, 4-point bend, flexural load was applied to the material at temperatures of 843°C.

Microstructural Analysis

In order to determine whether microstructural changes occurred within the filter matrices as a result of operation in the PCFBC process gas environment, sections of material were removed from each filter element at the conclusion of Test Segment 2 and Test Segment 3. Each section was fresh fractured and subjected to SEM/EDAX analyses. Characterization of each matrix was conducted along the outer membrane surface, pulse cycled surface, and throughout the cross-sectioned wall of the PCFBC-exposed filter elements.

In addition to information pertaining to the microstructure of each PCFBC-exposed filter element, SEM/EDAX analyses were utilized to determine the location of the ash and sorbent fines within the various filter materials. These analyses indicated that ash or sorbent fines were retained along the outer surface of the Schumacher Dia Schumalith FT20 and Pall 326 monolithic filter elements, with limited penetration into the barrier filter membrane. Penetration of fines was similarly limited to the first pore layers of the monolithic Coors P-100A-1 alumina/mullite filter elements. In contrast, fines were detected within the filtration mat of the 3M CVI-SiC composite candles. During pulse cycling, penetration of material (i.e., ash/sorbent fines after failure of an element; oxidation or metal wastage of structural components downstream of the filter array) typically to depths of 1-2 mm into each filter matrix resulted along the i.d. bore of the monolithic candles.

Schumacher Dia Schumalith FT20 Candle Filters

The as-manufactured, 10 mm thick structural support wall of the monolithic Schumacher Dia Schumalith FT20 filter matrix consisted of silicon carbide grains that were bonded together via a high temperature creep resistant binder. An ~100 μm thick membrane layer was applied to the outer surface of each filter element. The membrane consisted of ceramically bonded alumina fibers, as well as fine grained silicon carbide.

Extensive crystallization of the high temperature creep resistant Schumacher Dia Schumalith FT20 filter matrix occurred after 540 hours of operation in the 850°C PCFBC environment. Negligible changes were detected within the fibrous outer membrane.

In contrast after 1166 hours of operation in the PCFBC environment, an amorphous or melt-like phase resulted along the surface of the silicon carbide grains in the support matrix near the o.d. surface of the filter element. At the center of the cross-sectioned filter wall and i.d. surface, extensive crystallization of the coating that encapsulated the silicon carbide grains was evident. Although the binder-containing elements (i.e., aluminum, potassium, and sodium) were typically present within the silicon-oxygen-enriched layer that encapsulated the silicon carbide grains in the Schumacher Dia Schumalith FT20 filter matrix, in several areas, the concentration of these elements was relatively low. This implied that oxidation of the silicon carbide grains resulted, leading to the formation of SiO_2, and subsequently a reduction in the concentration of the original binder-containing elements within the layer that encapsulated the support grains.

Pall 326 Candle Filters

Similar to the Schumacher Dia Schumalith FT20 candle filters, the as-manufactured monolithic Pall 326 barrier filters consisted of silicon carbide grains that were bonded together via a high temperature creep resistant binder. A finer grained silicon carbide layer was applied to the outer surface of the 10 mm structural support wall of each filter element, forming an external membrane.

Extensive crystallization of the high temperature creep resistant binder resulted within the Pall 326 filter matrix after 540 hours of operation in the 850°C PCFBC environment. Changes within the fine grained silicon carbide membrane tended to indicate the presence of silica, implying that oxidation of the grit most likely occurred.

After 1166 hours of operation in the PCFBC environment, the Pall 326 filter matrix experienced extensive crystallization throughout the filter wall. The crystallized outer coating that encapsulated the silicon carbide grains contained areas that were enriched primarily with silica, while alternate areas predominantly contained mullite-like rod formations. Crystallization along the outer surface, and through the ~7 μm coating was evident. Along alternate grains near the o.d. surface of the 1166 hour, PCFBC-exposed, Pall 326 filter element, an amorphous silica-enriched encapsulating layer was identified.

Along the i.d. or pulse cycled surface of the 1166 hour, PCFBC-exposed, Pall 326 filter element, extensive crystallization of the encapsulating shell that coated the silicon carbide grains was clearly evident. Melt-like features of the silica-enriched outer coating were also observed. Below the encapsulating layer, the surface texture of the underlying silicon carbide grains was mottled resulting from what was considered to be surface oxidation of the grains during extended operation in the PCFBC environment.

Coors P-100A-1 Alumina/Mullite Candle Filters

In contrast to the clay bonded silicon carbide filter elements, the Coors P-100A-1 alumina/ mullite filter matrix consisted of mullite rods that were embedded within an amorphous phase that contained corundum (Al_2O_3) and anorthite ($CaAl_2Si_2O_8$). The monolithic Coors P-100A-1 alumina/ mullite filter elements were manufactured without an external surface membrane.

Both newly manufactured Coors P-100A-1 alumina/mullite candles, and candles that had been operated in the Westinghouse APF system at AEP were installed prior to initiating PCFBC testing in Test Segment 1 in the Foster Wheeler PCFBC test facility in Karhula, Finland. Post-test SEM/EDAX characterization of the 505 hour PCFBC-exposed, and 1650 hour PFBC/PCFBC-exposed Coors P-100A-1 alumina/mullite filters indicated that crystallization resulted along the pore cavity walls, and throughout the structural ligaments of the filter matrix.

At the conclusion of Test Segment 3, extensive crystallization was again observed along the pore cavity surfaces, and throughout the ligaments in the 1166 hour, PCFBC-exposed, and 2276 hour, PFBC/PCFBC-exposed, Coors P-100A-1 alumina/mullite filter matrices. Dendritic-like mullite rod formations protruded into the pore cavities, a fine grain phase frequently formed near the surface of the mullite rods, and a larger grain aluminosilicate phase formed along the surface of the pore cavities. In addition, a 3-4 μm nearly spherical aluminosilicate phase formed at the tip of the mullite rods primarily along the pulse cycled surface of the Coors P-100A-1 alumina/mullite filter matrix.

3M CVI-SiC Composite Candle Filters

The 3M CVI-SiC composite filter elements consisted of three layers — an open mesh, outer confinement layer, a middle filtration mat layer, and an inner structural support triaxial braided layer. Within the confinement and filtration mat layers, an ~1-2 µm layer of silicon carbide was chemically vapor infiltrated along the surface of Nextel™ 312 or alumina-based fibers, while an ~100 µm layer of silicon carbide was deposited along the Nextel™ 312 triaxial braid in the support matrix.

When removed from the filter array after 387 hours of operation at 850°C in the PCFBC environment, a color change was readily evident along the outer confinement and possibly filtration mat layers of the 3M CVI-SiC composite candle filters. The original dark black color of the CVI-SiC coating that encapsulated either the Nextel™ 312 fibers in the outer confinement layer, or the alumina-based fibers in the filtration mat layer was not retained along the majority of the filter body. Instead after 387 hours of operation, the outer confinement layer appeared to be white (i.e., excluding the presence of ash fines), while the filtration mat layer was generally a light to medium grey. The initial consideration was that "bare" fibers were present in the outer confinement layer as a result of removal of the CVI-SiC encapsulating shell during exposure of the 3M CVI-SiC composite filter elements to the high temperature oxidizing environment. Generally the triaxial support braid which consisted of an ~100 µm CVI-SiC layer that encapsulated twisted Nextel™ 312 filaments or fiber bundles, retained its as-manufactured dark black appearance.

SEM/EDAX characterization of the PCFBC-exposed 3M CVI-SiC composite filter matrix confirmed that removal of the SiC layer which initially encapsulated the Nextel™ 312 fibers in the outer confinement layer had occurred. Characterization of the lapped filtration mat indicated that oxidation of the 2 µm CVI-SiC shell had also occurred. As a result, an ~1 µm oxygen-enriched layer formed along the outer surface of the as-manufactured CVI-SiC coating. An oxygen-enriched region also formed along the inner surface of the CVI-SiC shell, bonding the shell to the filtration mat fibers. Bonding of the oxygen-enriched CVI-SiC shell to the fibers ultimately reduced the fracture toughness and possibly increased the strength of this layer.

Characterization of the triaxial support braid identified oxidation and pitting along the outer surface of the CVI-SiC shell that encapsulated the underlying Nextel™ 312 filaments. Within the interior of the filament or fiber bundles, a thin layer of CVI-SiC generally coated individual fibers. Frequently gaps were evident between the thin CVI-SiC layers and the fibers, as well as areas which clearly showed bonding of the shell to the surface of the contained fibers.

Gravimetric Analysis

During post-test inspection of the 1166 hour, PCFBC-exposed, filter elements, the candles were visually inspected, and the overall lengths of the filter elements were measured. Cracks were not observed along the outer surface of the filter body (i.e., below the flange of the clay bonded silicon carbide candles). An elongation of 9 mm was observed for the S350F/30

Schumacher Dia Schumalith FT20 candle, and between 6-8 mm of elongation was identified for the Pall 326 filter elements (Figure 5).

In order to discern whether oxidation of the silicon carbide grains was responsible for the resulting elongation or volume expansion of the clay bonded silicon carbide filter elements, Schumacher Dia Schumalith FT20 and Pall 326 filter samples were subjected to gravimetric analysis to determine the percent oxide that was present within each filter matrix. The results of the gravimetric analyses indicated that the as-manufactured Pall 326 filter matrix initially contained less free silica in comparison to the as-manufactured Schumacher Dia Schumalith FT20 filter matrix (Table 7). Operation in the PCFBC environment tended to enhance the concentration of silica within both clay bonded silicon carbide filter matrices.

For both Schumacher and Pall filter materials, a greater concentration of silica appeared to be present after 540 hours of PCFBC operation in comparison to the concentration of silica present after 1166 hours of operation. The 1166 hour, PCFBC-exposed, Schumacher and Pall filter matrices may have oxidized to a greater extent than the 540 hour, PCFBC-exposed matrices, but much of the newly formed SiO_2 was expected to have crystallized, forming cristobalite. Cristobalite is much more resistant to HF attack and thus, it was not detected by the gravimetric analysis technique.

High Temperature Oxidation vs Creep

Based on the gravimetric analyses which identified the increased silica concentration in the field-exposed filter materials, and the bench-scale, high temperature, flexural creep tests which identified negligible creep strain in the as-manufactured and field-exposed filter materials, oxidation of the silicon carbide matrix was considered to be the primary mechanism that was responsible for the 6-9 mm of elongation that was observed for the 1.5 m Schumacher Dia Schumalith FT20 and Pall 326 filter elements after 1166 hours of PCFBC operation. During oxidation of the silicon carbide grains, SiO_2 formed resulting in a volume expansion of the matrix (Figure 6). Continued operation of the previously PCFBC-exposed Schumacher and Pall filter elements is recommended to determine the rate at which oxidation/ elongation/creep occurs within the clay bonded silicon carbide materials as a function of extended plant operating time.

During PCFBC operation, consideration should also be given to competing volatilization mechanisms that release $Si(OH)_4$ from the silicon carbide filter matrix in the presence of steam. In contrast to oxidation, volatilization would be expected to reduce the overall volume of the silicon carbide grains.

Ash Characterization

Scanning Electron Microscopy/Energy Dispersive X-Ray Analysis

During operation in Test Segments 1 and 2 at the Foster Wheeler PCFBC test facility in Karhula, Finland, Illinois 6 coal and Linwood limestone were used as feed materials. Samples of the ash cake deposits which formed at various locations in the Westinghouse APF at the

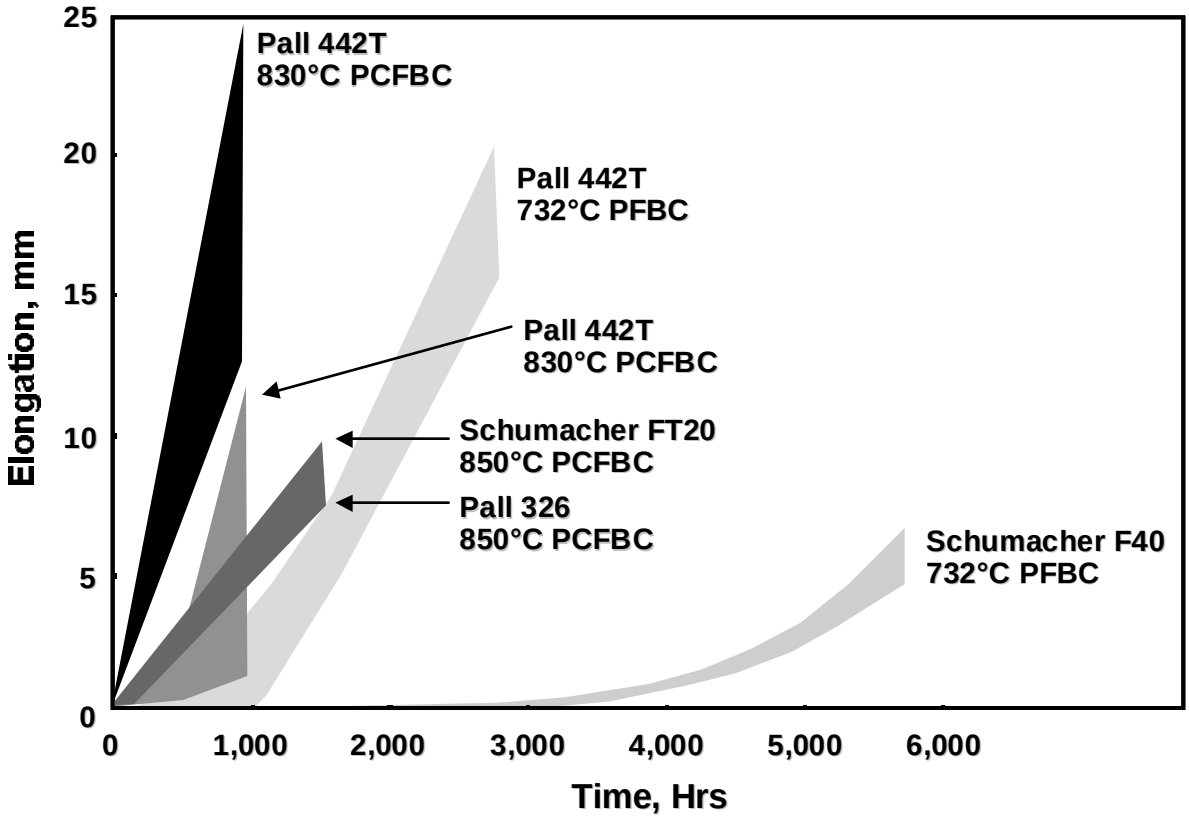

Figure 5 — Elongation of clay bonded silicon carbide candle filters.

Table 7
Silica Concentration in the As-Manufactured and PCFBC-Exposed Clay Bonded Silicon Carbide Candle Filter Materials

Filter Designation	Operating Time, Hrs	Percent Silica
Schumacher Dia Schumalith FT20		
S-350/108	—	11.72
S-350F/8 (T12)	540	17.23
S-350F/30 (T26)	1166	15.51
Pall 326		
R3-676	—	6.86
R5-665 (M21)	540	13.75
R6-674 (M26)	1166	9.77

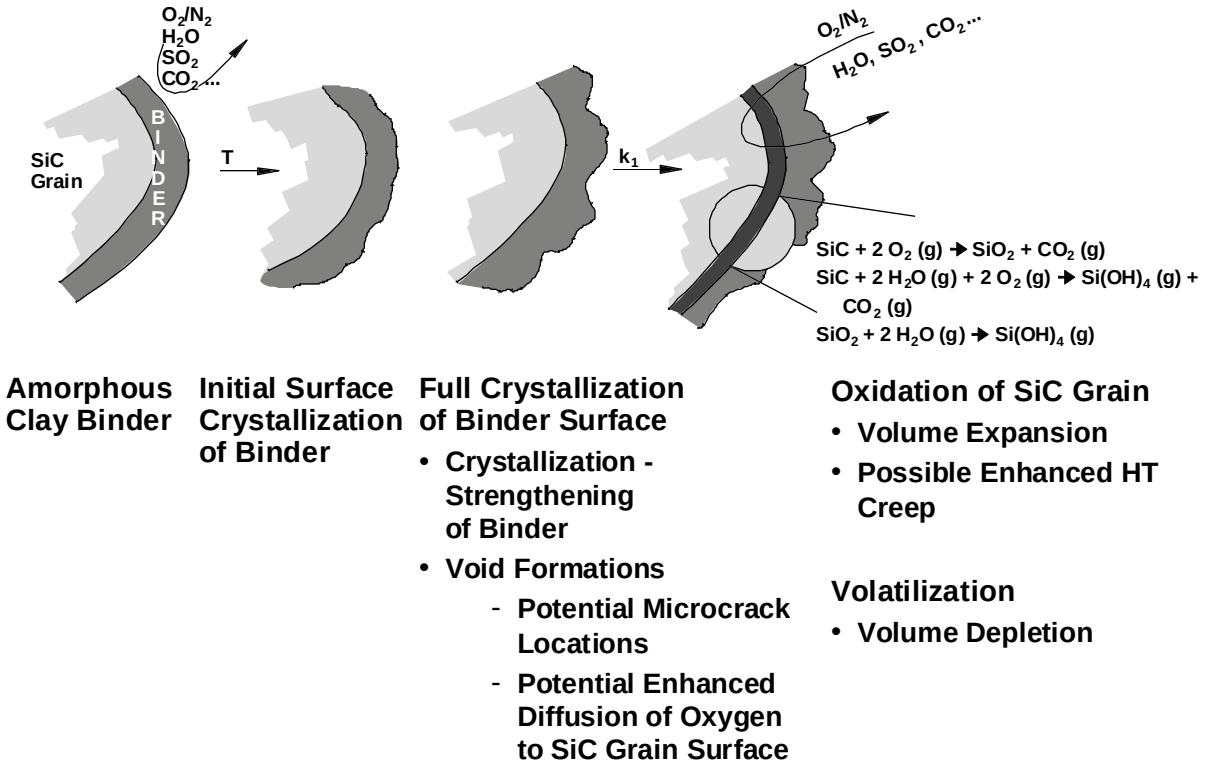

Figure 6 — Microstructural changes within the clay bonded silicon carbide candle filters during extended operation in PFBC/PCFBC process gas environments.

conclusion of Test Segment 2 were removed and subjected to SEM/EDAX analyses. Typically two types of particles were identified to be present within the ash cake layer that deposited along the outer surface of the candle filters. These included

- Submicron and micron particulates that were enriched with aluminum and silicon. These particles were considered to be entrained fly ash fines that were released during the combustion of coal. Melt-like features were evident along the surface of the ash fines.
- Larger sulfated or partially sulfated calcium-based sorbent fines.

The aluminum and silicon-enriched coal ash fines were generally seen to adhere to the larger sulfated or partially sulfated sorbent fines. Extensive crystalline features were evident along the outer surface of the sorbent particles. Agglomeration of crystalline formations and/or melt-like features were detected, particularly along the surface of coal ash fines. In addition,

spherical, ~10 μm particles enriched with silicon and calcium were also present within the deposited ash cake layer.

Dense packing of fines resulted within the ash cake layer that formed along the outer surface of the candle filters. Based on the analyses conducted, the cross-sectioned ash cake deposit did not show evidence of fracturing of bonds or melt-like phases that may have originally been present between adjacent particles. This implied close packing of fines, with limited point contact between particles prior to fresh fracturing of the deposited ash cake layer.

Ash deposits that collected along the metal filter holders were also characterized. SEM/EDAX analysis indicated that porosity existed within the interconnective network of ash and sorbent particles that deposited around the metal filter holders. Within the deposit, a melt-like phase formed, sintering adjacent particles together. The sintered bonds frequently formed point contact necks and channels between particles within the PCFBC deposit. The bonds were identified by EDAX to be silicon and/or silicon-aluminum-enriched.

Sorbent particles were also present within the ash cake deposit that formed around the metal filter holders. Based on EDAX analyses, the limestone sorbent was considered to be completely or nearly completely sulfated. As in the filter cake deposit, the metal holder deposit contained discrete sorbent fines which were larger than the retained ash fines or agglomerates.

Similar analyses were conducted on ash removed from the filter hopper. A greater quantity of the larger calcium-containing sorbent and possibly the iron-enriched particles was detected in the ash hopper deposit in comparison to the quantity of these particles found within either the filter ash cake layer or metal holder deposits. When viewed at high magnification, the filter hopper ash was seen to also contain extensive porosity. Porosity resulted from the interconnective network of fines that were held together through a silicon and/or aluminum-silicon-enriched melt-like point contact neck or channel phase.

X-Ray Diffraction Analysis

X-ray diffraction (XRD) analyses were conducted on select PCFBC dust cake deposits that formed at the conclusion of Test Segment 2. As shown in Table 8, the PCFBC ash which was generated during the combustion of Illinois coal and Linwood limestone consisted primarily of anhydrite and quartz, with secondary contributions of hematite, aluminosilicate phases, and an amorphous phase.

In order to determine whether the bulk composition of the filter cake changed when Iowa Industrial limestone and resized Linwood limestone were used in Test Segment 3, additional XRD analyses were conducted. As shown in Table 9, both the residual dust cake layer that remained along the surface of the candle filters, and ash bridged materials consisted principally of calcium sulfate with quartz and hematite as nearly equivalent secondary phases. Additional feldspar and pyroxene phases were present within the dust cake layer that formed along the

Table 8
XRD Analyses of the PCFBC Ash Deposits, wt%
— Test Segment 2 —

Sample Identification[(a)]	Anhydrite ($CaSO_4$)	Hematite (Fe_2O_3)	Quartz (α-SiO_2)	Alumino-silicate[(b)]	Amorphous Phase(s)	Unknowns
Candle T25 Concave Surface Of Ash Deposit	46	11	40	Trace	Small	3
Candle T25 Mid Section Of Ash Deposit	51	9	34	4	Small	2
Candle T25 Convex Surface Of Ash Deposit	51	9	33	4	Small	3
Ash Hopper Deposit Outer Surface	60	9	21	7	Small	3
Ash Hopper Deposit Mid Section	53	10	25	8	Small	4
Top Array Holder Deposit Outer Surface	57	9	27	4	Small	3
Top Array Holder Deposit Mid Section	60	9	28	3	Small	0

(a) Illinois coal and Linwood limestone deposit.
(b) Possible phases include: $CaAl_2Si_2O_8$ (Anorthite); $CaAl_2(Si_2OAl_2)O_{10}(OH)_2$ (Margarite); Al_2SiO_5 (Kyanite).

Table 9
XRD Analyses of the Candle Filter Residual Dust Cake Layer and Bridged Ash Materials
— Test Segment 3 —

Identified Phase[(a)]	Residual Candle Filter Surface Dust Cake Layers, wt%			Ash Bridges, wt%		
	T26	M26	B31	Top Plenum	Middle Plenum	Bottom Plenum
Calcium Sulfate ($CaSO_4$)	58.0	55.0	52.6	56.2	49.8	55.3
Quartz (SiO_2)	12.4	11.3	12.8	11.5	11.9	15.8
Hematite (Fe_2O_3)	11.0	12.0	12.0	10.8	8.0	11.2
Feldspar/ Plagioclase $(Ca,Na)(Al,Si)_2Si_2O_8$	9.5	9.5	10.9	10.6	11.7	7.6
Pyroxene/Clinopyroxene $(Ca(Mg,Fe^{+3},Al)(Si,Al)_2O_8)$	9.0	12.1	11.7	11.0	18.6	10.2

(a) Illinois coal and Linwood limestone/Iowa Industrial limestone/Resized Linwood limestone deposit.

surface of the filter elements. Although major, these phases were present at concentrations that were slightly lower than the secondary quartz and hematite phases. In contrast, however, the concentration of the feldspar and pyroxene phases were either equivalent to or greater than the concentration of quartz and hematite within the various ash bridged materials. Based on the XRD analyses provided in Table 9, the concentration of calcium sulfate within the dust cake layer appeared to decrease as a function of array location (i.e., top > middle > bottom).

In view of the XRD analyses presented in Tables 8 and 9, a significantly reduced concentration of α-SiO_2 was identified in the PCFBC dust cake formations that resulted within the filter cluster assembly during Test Segment 3. In contrast a greater concentration of feldspars and/or aluminosilicates was identified in Test Segment 3's dust cake formations. The reduced concentration of α-SiO_2 present within the melt-like necks or channels between adjacent particles, and the increased concentration in the aluminosilicate phases were considered to contribute to limiting the extent of ash bridging during PCFBC operation in Test Segment 3.

Density, Moisture Content, and Compressive Strength

Room temperature compressive strength testing was conducted on fourteen ash deposits that were removed from the Westinghouse APF at the conclusion of Test Segment 2. Typically the deposits were cut into either cubic or rectangularly shaped samples prior to testing. In addition, the moisture content and bulk density of the samples were determined.

The density of the ash deposits ranged between 0.428 and 0.718 gm/cm^3, and the moisture content ranged between 0.02 and 0.07%. The room temperature compressive strength of the PCFBC ash deposits ranged between 0.7 and 34.4 psi. In addition,

- Variation in compressive strength was identified to exist within ash deposits formed at a given sample location.
- The compressive strength of the ash bridges that formed between filter elements was shown to be approximately equivalent to the compressive strength of the hopper ash deposit.
- The candle filter ash cake appeared to be weaker than the ash deposits that formed between adjacent filter elements, or between the filter elements and the plenum pipe.
- The dust shed deposits appeared to be weaker than the candle filter cake deposits.
- The compressive strengths of the filter holder deposits and isolated bridges that formed near the bottom of the filter elements were nearly identical (Table 10). The isolated bridges may have resulted from the collection of ash that had fallen from the metal holders.

The average density of all twenty-seven PCFBC ash samples was 0.587 g/cm^3 which was comparable to the density of the PFBC bridges that formed at the conclusion of Test Segments 3 and 4 at AEP. Clearly the density of the PCFBC ash deposits was less than the density of the deposits that formed within the i.d. bore of the filter elements (i.e., 1.19 g/cm^3), or along the metal filter holders along the top array at the conclusion of Test Segment 5 at AEP (i.e., 0.6-0.85 g/cm^3). Similar analyses were not conducted on the PCFBC ash formations that resulted within

Table 10
Room Temperature Compressive Strength of PCFBC Ash Deposits
— Test Segment 2 —

Location	Average Strength ± 1σ, psi (Number of Samples Characterized)
Filter Holders	4.9 ± 3.3 (5)
Bridges Between Candle Filters (Top, Middle, Bottom Locations)	15.4 ±7.6 (11)
Isolated Bridges At The Bottom Of The Filter Elements	4.4 ± 1.6 (3)
Candle Filter Cake	10.4 ± 6.8 (4)
Dust Shed	8.1 ± 4.2 (2)
Hopper	16.5 ± 1.7 (2)

the Westinghouse APF at the Foster Wheeler test facility in Karhula, Finland, at the conclusion of Test Segment 3.

Conclusions

The stability of the commercially available oxide-based Coors P-100A-1 alumina/mullite and the high temperature creep resistant clay bonded silicon carbide Schumacher Dia Schumalith FT20 and Pall 326 candle filters was demonstrated during 1166 hours of operation in the Foster Wheeler PCFBC test facility in Karhula, Finland. Both the Coors and Pall matrices experienced an initial loss of bulk material strength while undergoing numerous microstructural changes. In contrast the strength of the Schumacher matrix remained virtually unchanged while numerous microstructural changes were encountered.

Based on the analyses conducted in this program, oxidation of the clay bonded silicon carbide Schumacher Dia Schumalith FT20 and Pall 326 candle filters was considered to have occurred which promoted elongation of the elements during extended PCFBC operation. Oxidation, as well as removal of the oxidized CVI-SiC deposited layer resulted along the outer confinement and filtration mat layers of the 3M CVI-SiC composite filter matrix. A rapid reduction in bulk strength, leading to the general conditioning of the 3M CVI-SiC composite matrix subsequently resulted during operation in the PCFBC environment.

As a result of testing at Karhula, the oxide-based Coors P-100A-1 alumina/mullite candle filters continue to show promise for achieving extended PCFBC operating life in advanced coal-fired applications. Continued exposure of the Coors P-100A-1 alumina/mullite, Schumacher Dia Schumalith FT20, and Pall 326 candle filters in the PCFBC environment, followed by accelerated life cycle testing is recommended. Similarly monitoring the impact of oxidation of the clay bonded silicon carbide filter elements, and the associated elongation of the candles is essential to determine the long-term viability of the high temperature creep resistant Schumacher

Dia Schumalith FT20 and Pall 326 filter matrices. Development of an oxidation resistant coating along the surface of the silicon carbide grains, as well as throughout the 3M CVI-SiC composite filter matrix, or alternately manufacturing a complete oxide matrix/fiber composite candle filter may foster improved life of the fiber reinforced composite filter elements.

Characterization of the dust cake deposits which formed throughout the Westinghouse APF filter cluster identified calcium sulfate and quartz as primary phases, with hematite present as a secondary phase. Complex aluminosilicates were detected as minor phases within the ash cake deposits. The potential cohesivity or "stickiness" of the dust cake layer due to the presence of silica or silicates, the impact of particle size within the various deposits, and the relationship of the coal feed and sorbent composition on the resulting dust cake chemistry are areas which warrant continued investigation in order to achieve viable commercial operation of advanced hot gas filtration systems.

Qualification Testing of Advanced Oxide-Based Monolithic and Composite Candles

Although the Coors, Schumacher, and Pall surveillance candles remained intact during the entire 1995-1996 test campaign in the Westinghouse APF at the Foster Wheeler test facility in Karhula, Finland, issues remain regarding the long-term viability of these materials. These issues include thermal fatigue of the monolithic oxide-based Coors P-100A-1 alumina/mullite filter matrix, and elongation, oxidation, creep and/or crack growth of the clay bonded silicon carbide Schumacher Dia Schumalith FT20 and Pall 326 filter materials. Alternate manufacturers have been developing advanced oxide-based monolithic and composite filters in attempts to avert many of the projected long-term issues that potentially could limit the operational life of the hot gas filtration system.

Prior to demonstrating extended filter operating life, demonstrating retrofit capability into existing hardware and acceptable performance under a rigorous qualification test program has been considered by Westinghouse as a critical activity which significantly reduces the initial risk of failure when newly developed filters are installed and tested in the field. Early in 1997, Westinghouse provided the following suppliers with the candle filter dimensional tolerances, as-manufactured gas flow resistance, and filter material properties that are typically specified for supply of commercial candle filter elements:

- 3M — Oxide-based Nextel™ 550/720 elements
- B&W — Oxide-based vacuum wound Nextel™ 610 elements
- Techniweave — Oxide-based continuously woven 2D Nextel™ 720 or Nextel™ 610 elements
- DuPont — PRD-66 elements (Coarse and medium grade membrane)
- Blasch — Mullite bonded alumina elements
- Specific Surface — Taperflow™ monolithic elements
- Scapa — Cerafil vacuum infiltrated fibrous elements.

All candles that were provided for use in the qualification program were subjected to a preliminary inspection, identifying overall dimensional tolerances and room temperature gas flow

resistance (Figure 7). One candle from each of the above filter suppliers was then installed in the Westinghouse PFBC simulator test facility. In addition, a newly manufactured Coors P-100A-1 alumina/mullite candle, and a 1110 hr, PFBC-exposed, Coors P-100A-1 alumina/mullite candle were installed in the array. The filter array was initially subjected to ~120 hours of steady state operation utilizing reentrained PFBC ash fines. All elements remained intact during this segment of qualification testing.

In order to simulate the impact of possible long-term thermal fatigue or shock on the stability of the various filter matrices, the filter array was subsequently subjected to ~2200 accelerated pulse cleaning cycles, and 12 thermal transient cycles. Shear failure of the Techniweave Nextel™ 610 flange occurred after five thermal transient events. Modification of the flange configuration and reduction in the gas flow resistance of the as-manufactured filter elements were recommended prior to installation and operation of the Techniweave candles in the field.

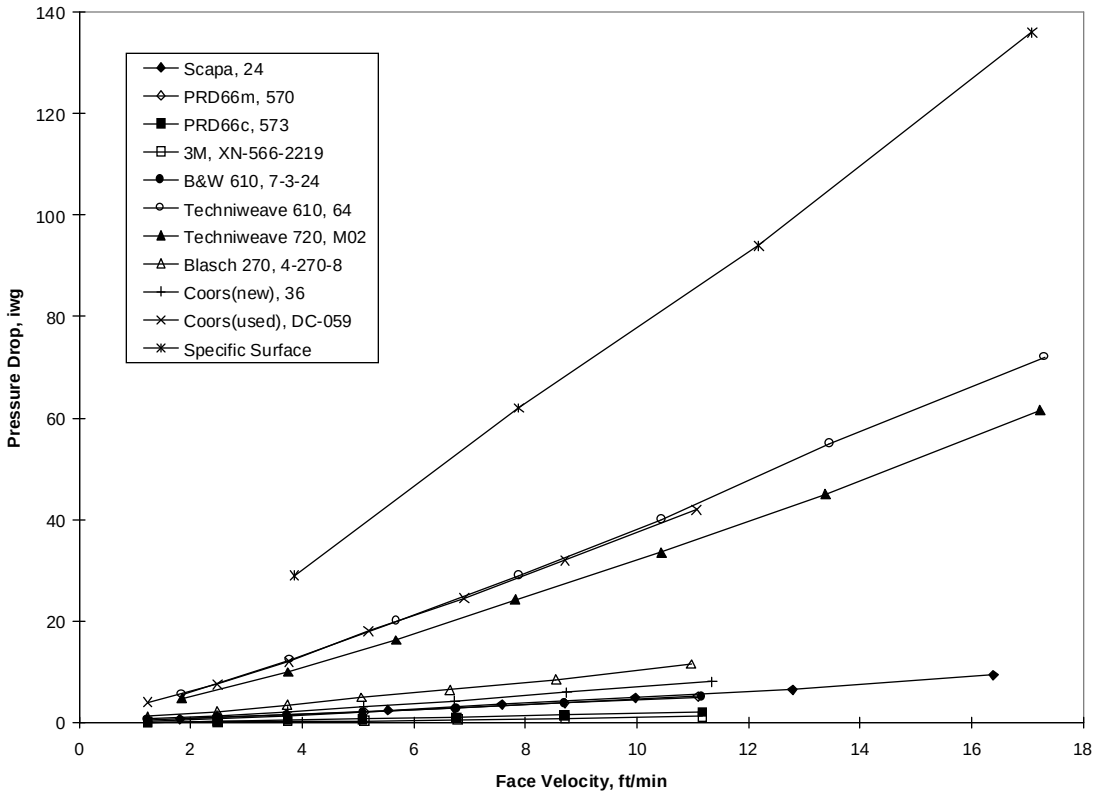

Figure 7 — Room temperature gas flow resistance of the as-manufactured advanced monolithic and composite candle filters prior to qualification testing in the Westinghouse PFBC simulator test facility.

Post-test inspection of the filter array was conducted which included

- Identification of the ease of removal of the elements from the metal holders
- Assessment of the primary and secondary gasket seal performance
- Assessment of the integrity of the flange, outer membrane, filter body, and closed end cap
- Conduct of room temperature gas flow resistance measurements.

As shown by the process temperature c-ring compressive strength information presented in Figure 8, the residual bulk strength of the B&W, Blasch, 3M, Techniweave 720, and newly manufactured Coors P-100A-1 matrices tends to decrease as a result of the elements being subjected to the simulated PFBC test conditions. Microstructural characterization and phase composition analyses are expected to provide insight into the mechanisms that lead to the reduction in strength of both advanced monolithic and composite filters.

As previously demonstrated by Westinghouse, the bulk strength of the DuPont PRD-66 matrix tends to increase during simulated or field exposure.[1] This was considered to result from the bulk vs barrier filtration characteristics of the material, whereby submicron and micron fines penetrate through the membrane of the DuPont PRD-66 filter element and become entrapped within the filter wall. Although divot formations along the outer membrane did not occur during the qualification test program, the potential may still exist during extended field operation particularly if thermal expansion of the ash fines within the wall occurs during plant startup cycles,[3] or hydration of the ash results during shutdown cycles.

In addition to the material properties developed by Westinghouse for the advanced monolithic and composite filter materials (Table 11), SEM/EDAX and XRD analyses of the as-manufactured and qualification-tested filters are being conducted. With extended operation of the advanced monolithic and composite filter elements in the actual PCFBC environment, the response of the various porous ceramic matrices (i.e., crystallization of the monoliths; retention of the fracture toughness properties or embrittlement of the fibrous composites; etc.), and the overall component stability will be determined.

Candle Filter Exposure in IGCC Applications

In order to assess the stability of the advanced filter materials during long-term operation in IGCC applications, Westinghouse has designed a mini-filter system which is tentatively planned to be installed and operated at the Sierra Pacific Power Company, Piñon Pine Demonstration Plant in Reno, Nevada. The mini-filter system will be located downstream of Westinghouse's main APF system which currently houses 784, 1.5 m Pall Vitropore 442T filter elements.

In the mini-filter, porous ceramic and sintered metal mini-candles (i.e., 305 mm) will be exposed to cleaned, particulate-free, fuel gas for extended periods of time. In addition to the ceramic filter elements, alternate ceramic filter material and structural metal coupons will be

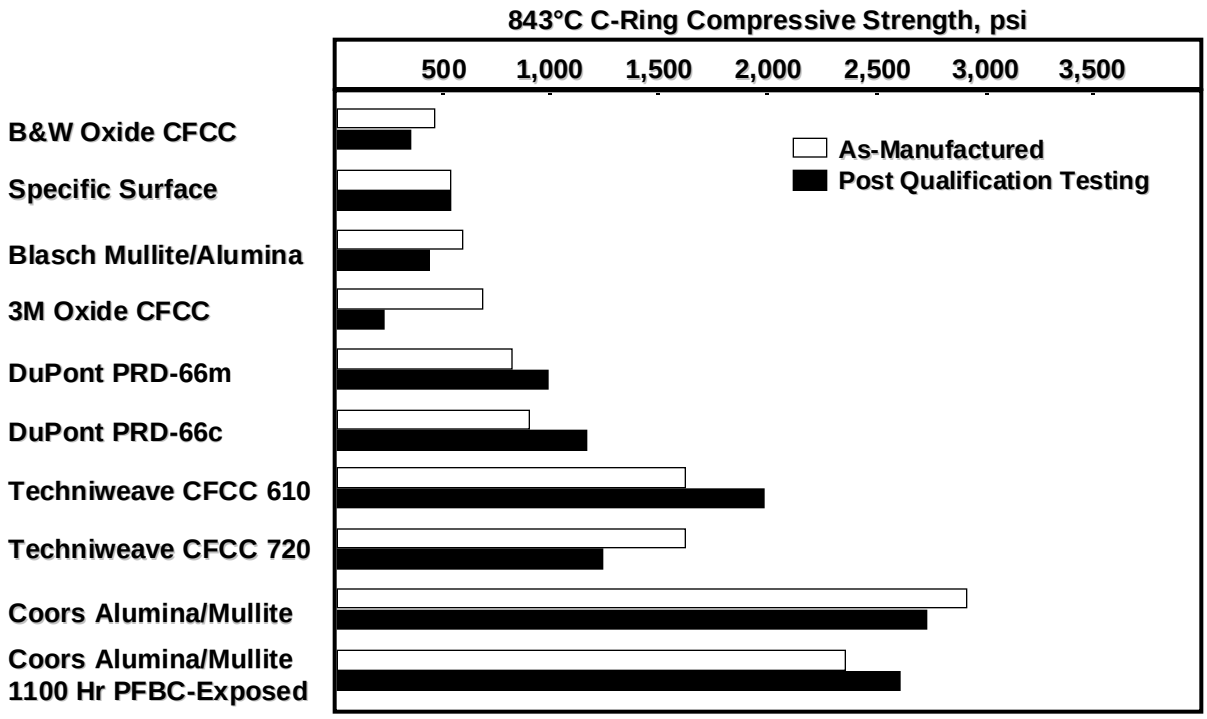

Figure 8 — Bulk strength of the as-manufactured and qualification-tested advanced monolithic and composite candle filters.

housed in the mini-vessel and exposed to the fuel gas environment in a flow-over fashion. To date, Westinghouse has purchased and inspected the following mini-candles:

- Schumacher Dia Schumalith T10/20
- Schumacher Dia Schumalith FT20
- Pall 326
- Pall Vitropore 442T
- Coors P-100A-1 Alumina/Mullite
- DuPont PRD-66
- 3M CVI-SiC
- Pall Iron Aluminide.

Table 11
Material Properties of the As-Manufactured and Qualification Tested Advanced Monolithic and Composite Candle Filters

Candle Identification Number	Status	Burst Pressure, psi	Ultimate Hoop Stress, psi	Modulus, psi x 10^6	Poisson's Ratio
DuPont PRD-66 (Coarse Membrane)					
D-563c	As-Manufactured	148	555	7.96	0.86
D-573c	Post-Test	158	597	6.11	0.82
DuPont PRD-66 (Medium Membrane)					
D-564m	As-Manufactured	180	691	7.09	0.84
D-570m	Post-Test	170	653	5.42	0.84
B&W Oxide-Based Composite					
7-3-21	As-Manufactured	188	998	1.25	0.95
7-3-24	Post-Test	136	776	1.17	0.85
3M Oxide-Based Composite					
XN-566-2214	As-Manufactured	52	586	1.36	0.73
XN-566-2219	Post-Test	29	334	1.35	0.73
Techniweave Oxide-Based Composite (Nextel™ 610)					
T-65	As-Manufactured	ND	ND	ND	ND
T-64	Post-Test	ND	ND	ND	ND
Techniweave Oxide-Based Composite (Nextel™ 720)					
T-M01	As-Manufactured	ND	ND	ND	ND
T-M02	Post-Test	ND	ND	ND	ND
Blasch Mullite Bonded Alumina					
B-4-270-3	As-Manufactured	170	410	2.12	0.09
B-4-270-8	Post-Test	155	376	1.60	0.09
Specific Surface Taperflow™					
SS-0	As-Manufactured	ND	ND	ND	ND
SS-1	Post-Test	ND	ND	ND	ND
Scapa Cerafil					
S-24	As-Manufactured	ND	ND	ND	ND

ND: Not determined.

Westinghouse has designed the mini-filter vessel and ancillary equipment, meeting design specifications identical to those required for design and construction of the main vessel. Currently Westinghouse is awaiting approval by M. W. Kellogg and Sierra Pacific for acceptance of the design packages, and M. W. Kellogg's interface effort for installation of the mini-filter system into existing plant process gas stream lines. Once the mini-filter vessel and ancillary equipment design packages are accepted by M. W. Kellogg and Sierra Pacific, Westinghouse will initiate the manufacture of the mini-filter vessel and ancillary equipment, prior to shipment and installation at site.

Application/Benefits

As a key component in advanced coal- or biomass-based power applications, hot gas filtration systems protect the downstream heat exchanger and gas turbine components from particle fouling and erosion, cleaning the process gas to meet emission requirements. When installed in either PFBC or IGCC plants, lower downstream component costs are projected, in addition to improved energy efficiency, lower maintenance, and elimination of additional and expensive fuel or flue gas treatment systems. As a critical component, long-term performance, durability, and life of the porous ceramic filter elements are essential to the successful operation of the hot gas filtration system in advanced combustion and gasification applications.

Future Activities

Efforts will be focused on

- Assessment of the microstructural and phase changes that resulted within the advanced monolithic and composite filter elements during qualification testing under simulated PFBC operating conditions.

- Introduction and exposure of the advanced monolithic and composite filter elements to PCFBC operating conditions in the Westinghouse APF at the Foster Wheeler test facility in Karhula, Finland.

- Continued PCFBC exposure of commercially available oxide and nonoxide-based monolithic filter elements in the Westinghouse APF at the Foster Wheeler test facility in Karhula, Finland.

- Characterization of the PCFBC-exposed commercially available and advanced monolithic and composite surveillance filter elements.

- Conduct of accelerated life testing of field-exposed filter elements and development of life assessment models.

- Construction, installation, and operation of the mini-filter system at the Sierra Pacific Power Company, Piñon Pine, IGCC test facility in Reno, Nevada.

- Characterization of the IGCC-exposed ceramic and sintered metal filters and structural metal coupons.

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